

**EFFECT OF SAWDUT SPECIES AND PARTICLE SIZE ON XYLOSE
PRODUCTION**

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of the requirements for the award of the degree of
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ABSTRACT

Xylose is a pentose sugar and commonly hydrolyzed from lignocellulosic material. This study was carried out to identify the species and particle size on xylose production was investigated. Three types of hardwood species (*Meranti*, *Keruing* and *Resak*) and five differences particle size of sawdust (800, 615, 400, 315 and 200 μm) were used in the production of xylose. Hydrolysis method by using diluted sulfuric acid was employed. The sawdust was reacted with diluted sulfuric acid to degraded the hemicellulose from lignocellulosic structure. The higher xylose concentration that produced was 32.12 g/l. The overall results indicated that the sawdust species and particle size were *Keruing* and 400 μm respectively exhibited the highest concentration of xylose due to the acid hydrolysis effects. From the Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscope (SEM), the structure of treated sawdust was change. The maximum xylose production from sawdust was produce at 3.24% sulfuric acid concentration and reaction time 60 minutes.

ABSTRAK

Xylosa adalah gula pentosa dan biasanya dihidrolisiskan daripada sisa buangan tumbuh-tumbuhan. Kajian ini dijalankan bertujuan untuk mengenalpasti spesis dan saiz habuk kayu yang terbaik dalam penghasilan xylosa telah dikaji. Tiga jenis spesis kayu keras (*Meranti*, *Keruing* dan *Resak*) dan lima saiz habuk kayu (800, 615, 400, 315 dan 200 μm) digunakan dalam penghasilan xylosa. Kaedah hidrolisis dengan menggunakan asid sulfurik cair telah bertindak balas dengan habuk kayu bagi menyingkirkan hemisellulosa daripada ikatan struktur selulosa. Kepekatan xylosa yang maksima terhasil adalah 32.12 g/L. Keputusan keseluruhan menunjukkan bahawa spesis dan saiz habuk kayu yang terbaik dalam penghasilan xylosa yang tinggi adalah *Keruing* dan 400 μm . Keputusan analisa daripada Spektrum Infra Merah (FTIR) dan imbasan Mikroskopi Elektron (SEM) menunjukkan perubahan struktur dan permukaan sisa habuk kayu selepas proses hidrolisis. Penghasilan maksimum xylosa daripada habuk kayu adalah pada kepekatan asid 3.24% dan masa prarawatan 40 minit.

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LIST OF ABBREVIATIONS & SYMBOLS

%	-	Percentage
wt%	-	Weight percent
g	-	Gram
kg	-	Kilogram
ml	-	Milliliter
μl	-	Microliter
CO ₂	-	Carbon Dioxide
H ₂ O	-	Water
°C	-	Degree Celsius
FTIR	-	Fourier Transform Infrared Spectroscopy
HPLC	-	High Liquid Performance Chromatography
OPF	-	Oil Palm Fronds
RMW	-	Red Meranti Wood
SEM	-	Scanning Electron Microscope

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CHAPTER 1

INTRODUCTION

1.1 BACKGROUND OF RESEARCH

There are four main sources of lignocellulosic materials which are forest biomass, herbaceous grass, agriculture residue and municipal waste. The production of lignocellulosic materials is increasing throughout the year in the peninsular Malaysia and the economical disposal of them is a serious problem to the sawmill industries. Bioconversion and hydrolysis of the lignocellulosic waste materials residue can produce a valuable product since it is renewable, widespread and cheap sources of raw material in nature and it is can become an environment friendly option to reducing generated of waste (Kuhad and Singh, 1993). Because of that, most of the industries increasing towards to reuse and recycle back the agro-industrial by-products to produce renewable product. It is commonly used as fuel in manufacturing plants and local utilities and chemicals or food ingredient production (Musatto *et al.*, 2006).

The majority of lignocellulosic materials are constructed from three major polymeric components which are cellulose, hemicellulose and lignin (Gabrielii *et al.*, 2000). Since xylose is present in small amounts in fruits and vegetables, the extraction from these sources is uneconomical. Commonly, chemical reduction of xylose is expensive because extensive purification and separation steps are necessary (Parajo *et al.*, 1996). However, there are others lignocellulosic waste material like wood, rice husk, corn stalk, wheat straw and flax straw that treated as a waste in the industry. There are present high amount of xylose compared to fruit and vegetables (Sjoman *et al.*, 2008).

In this research, hardwood species of sawdust is being used as a raw material in the production of xylose. Sawdust is a residue from the sawmill industries that usually treated as a waste. However, it is a heterogeneous material composed primarily of cellulose, hemicellulose, and lignin (Bludworth *et al.*, 1993). The cell wall polysaccharides in the sawdust can degraded into their corresponding constituents by hydrolytic procedures either hydrothermal process, enzymatic process or acidic process. On hydrolysis, cellulose yields glucose and the non-cellulosic polysaccharides yields xylose, mannose, galactose and arabinose as well as acetic and hydroxycinnamic acids (Musatto *et al.*, 2006).

1.2 PROBLEM STATEMENT

Nowadays, in Agro-industrial and plantation of timber industry, there is produced high value of hardwood saw mill residue, which is currently treated as solid waste (Nirdosha *et al.*, 2009). In practice this residue is burned in indicators which may be causes of environmental pollution problems in nearly localities and offers limited value to the industry (Rahman *et al.*, 2006). Furthermore, saw mill residue uses is still limited. Basically, it is used as animal feed or simply as landfills (Musatto *et al.*, 2006).

By considering this scenario, an alternative practice should be considered by the sawmill industry to commercialize the residue from hardwood species to recycle back without causing environmental pollution and produce valuable product. This practice will requires less energy, and diminishes pollutants in industrial effluents, as well as being more economically advantageous due to its reduced costs.

According to the prior research on the hydrolysis of saw mill residue, it is only focus on the softwood species such as corn, rice husk, sugarcane baggase and so on. However, there are lacked of studies regarding production of xylose from hardwood species of sawdust. The information regarding this sugar monomer that produced from the hardwood species of sawdust is also hardly available.

1.3 RESEARCH OBJECTIVE

- The main objective is :-
 - To produce maximum yield of xylose from three types of sawdust and differences particle size.
- The specific objective are :-
 - To determine the amount of xylose production in the difference species and particle size of hardwood.
 - To determine the effect of species and particle size in xylose production by qualitative and quantitative analysis.
 - To determine the best type of species and particle size of sawdust on xylose production.

1.4 SCOPE OF RESEARCH

- Focus on the production of xylose from hardwood species of sawdust (*Meranti*, *Keruing* and *Resak*) that are collected from saw mill industry at Gambang and Kuantan Pahang.
- Study the effect of five differences particle size of sawdust in xylose production (0.80, 0.63, 0.40, 0.315 and 0.20 mm).
- The qualitative analysis involve is Scanning Electron Microscope (SEM) and Fourier Transform Infrared Spectroscopy (FTIR).
- The quantitative analysis involved is High Performance Liquid Chromatography (HPLC) and Kappa Number method.

1.5 SIGNIFICANCE OF RESEARCH

Nowadays, there is great political and social pressure to reduce the pollution arising in industrial activities. Almost all developed and underdeveloped countries are trying to alter this reality by modifying their processes so that their residues can be recycled to produce more valuable product (Musatto *et al.*, 2006). Since wood sawmill residue is an economic and widespread resource in Pahang, Malaysia, processing of difference type of sugar monomer is the first successful commercial application with several research initiatives underway to find uses of sawmill residue (Nirdosha *et al.*, 2009).

Xylose is one of the sugar monomer that can be produced from hardwood species of sawdust. However, there are lacks of studies regarding the production of xylose from hardwood species of sawdust. Most of the prior studies are only focusing on the softwood species like rice straw, sugarcane baggase and sago trunk cortex. Other than that, there are no studies regarding the particle size and sawdust species of hardwood in the xylose production. Hence, this research is come out to focusing on the studies of the effect of particle size and sawdust species of hardwood in xylose production.

CHAPTER 2

LITERATURE REVIEW

2.1 OVERVIEW OF LIGNOCELLULOSIC BIOMASS

Malaysia is well known for its widespread renewable resources of agriculture, municipal waste and forest residue. It is commonly known as lignocelluloses biomass which highly potential to renewable, widespread, and a cheap source of the residue that can be used as a raw material to become marketable products such as biofuel, bioenergy, and added value biomolecules by using bioconversion process. Lignocellulosic materials are commonly come from four differences sources (Table 2.1) which are forest biomass, agricultural residue, herbaceous grass and municipal waste.

Table 2.1: Sources of Lignocellulosic material

Specification	Source	Example
Forest biomass	Wood	<ul style="list-style-type: none"> • Hardwood • Softwood
	Residue	<ul style="list-style-type: none"> • Bark • Thinning • Sawdust • Pruning
Agricultural residue	Food crop	<ul style="list-style-type: none"> • Corn • Stover • Kernel fibers • Wheat straw
	Non-food crops	<ul style="list-style-type: none"> • Cotton stalk • Sugarcane bagasse
Herbaceous grass	Grass	<ul style="list-style-type: none"> • Switch grass • Bermuda grass • Rye straw
Municipal waste	Residential source	<ul style="list-style-type: none"> • Waste paper • Waste food
	Non-residential	<ul style="list-style-type: none"> • Paper mill • Sludge waste paper & board

2.2 STRUCTURE OF LIGNOCELLULOSIC BIOMASS

Lignocellulose is a structural material in cell wall of plant that surrounded by a polysaccharide which provides support, strength and shape to the plant. It is commonly found in roots, stalks and leaves (Sierra *et al.*, 2006). It is complex internal structure and mainly composed of three major components which are cellulose, hemicelluloses and lignin. The component of the lignocellulosic biomass is shown in Figure 2.1. In the structure, Lignin provides structural function as a matrix in which cellulose and hemicellulose is embedded. While, cellulose obtained the crystalline fibrous structure and it appears to become the core of the complex. Then, Hemicellulose is positioned in between the microfibrils and macrofibrils of cellulose. The structure of the lignocellulose structure is shown in the Figure 2.2.

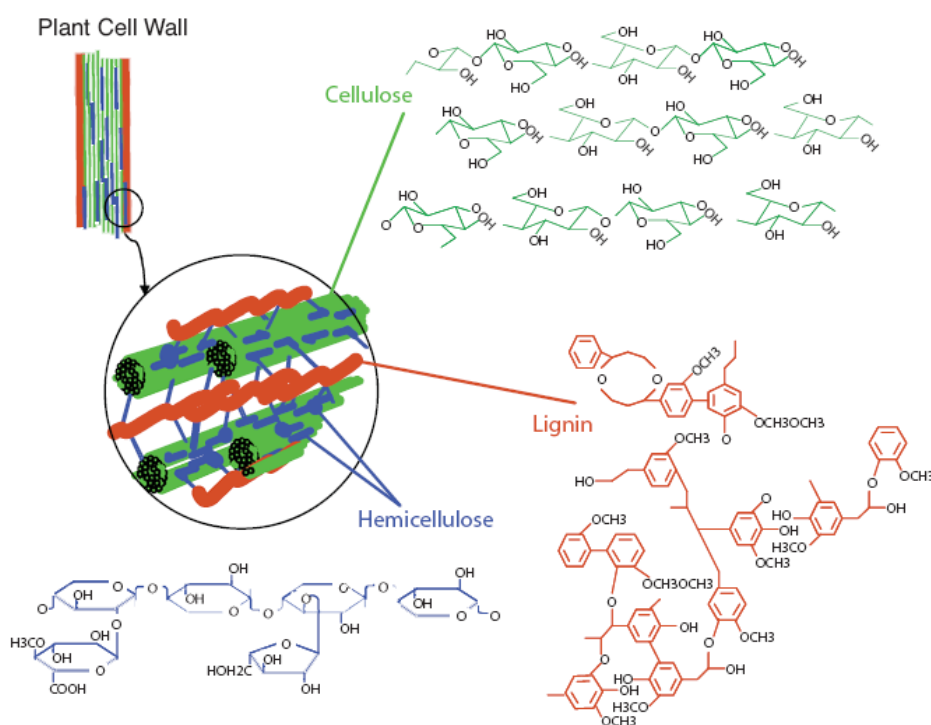


Figure 2.1: Component of lignocellulosic biomass

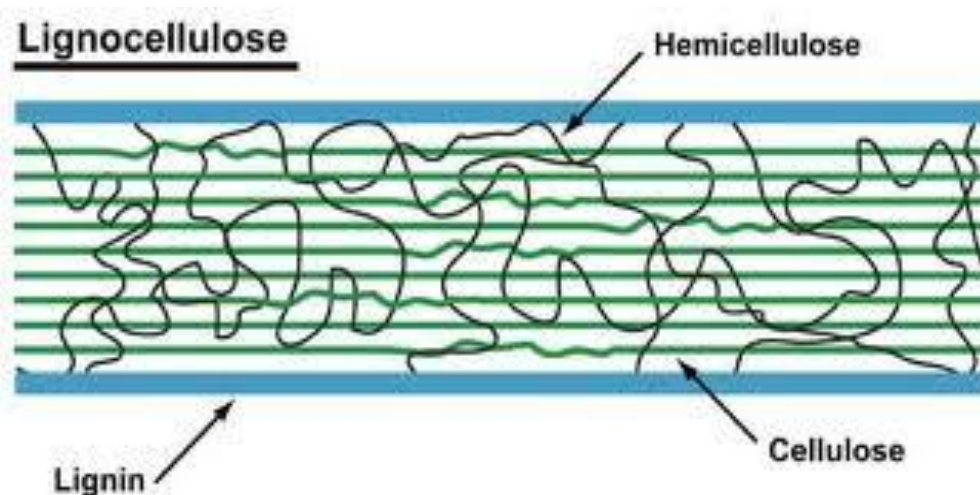


Figure 2.2: Structure of lignocellulose biomass

Furthermore, The structure of lignocellulose also consist of small amount of other component like water, protein and other compounds which do not participate significantly in the forming of lignocellulose structure. However, the content of the main components of the lignocellulose biomass in the nature are vary in the range shown in the Table 2.2.

Table 2.2: The contents of cellulose, hemicelluloses, and lignin in common agricultural residue and waste (Sun and Cheng, 2002)

Lignicellulosic materials	Cellulose (%)	Hemicelluloses (%)	Lignin (%)
Hardwood stems	40-55	24-40	18-25
Softwood stems	45-50	25-35	25-35
Nut shells	25-30	25-30	30-40
Corn cobs	45	35	15
Grasses	25-40	35-50	10-30
Paper	85-99	0	0-15
Wheat straw	30	50	15
Sorted refuse	60	20	20
Leaves	15-20	80-85	0
Cotton seed hairs	80-95	5-20	0
Newspaper	40-55	25-40	18-30
Waste papers from chemical pulps	60-70	10-20	5-10
Primary wastewater solids	8-15	NA	24-29
Swine waste	6.0	28	NA
Solid cattle manure	1.6-4.7	1.4-3.3	2.7-5.7
Coastal Bermuda grass	25	35.7	6.4
Switch grass	45	31.4	12.0

2.2.1 Lignin

Lignin is the most abundant natural non-carbohydrate organic compound located in lignocellulosic biomass structure. It is form by polymer of aromatic subunits with complex molecule and contains highly branches polymer that usually derived from phenyl propane units linked. There are three types of phenyl propionic alcohols that usually exist as monomers of lignin which are coniferyl alcohol, sinapyl alcohol, and coumaryl alcohol. Each of this monomer has an aromatic ring with different substituents (Mussato *et al.*, 2010). The linking of monomers that consisting a variety of bonds is shown in Figure 2.3.

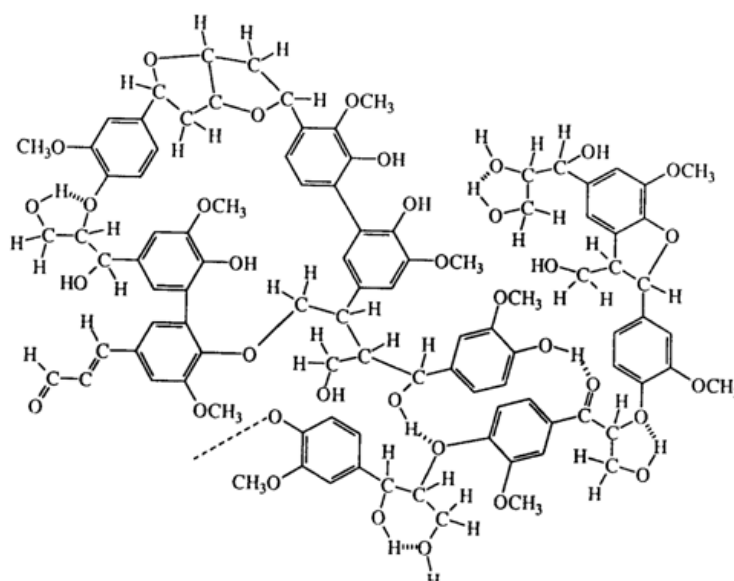


Figure 2.3: Structure of a section of a lignin polymer

In the lignocellulose, lignin is closely bound to cellulose and hemicellulose and able to form covalent bonds with hemicellulose. Due to the existing bond, it will provide structural rigidity and prevention of swelling of lignocelluloses.

2.2.2 Cellulose

Cellulose is predominant polymer in lignocellulosic biomass followed by hemicellulose and lignin. According one of the studies from Howard *et al.*(2003) shows that the cellulose composition in the hardwood and softwood species are higher than hemicellulose and lignin. It is form in linear homopolymer anhydro D-glucose unit linked together by β -1,4-polyacetal of cellobiose (4-O- β -D-glucopyranosyl-D-glucose) (Figure 2.4). Highly ordered crystalline regions are formed in the formation of hydrogen bonding between cellulose molecules. The crystalline molecules are formed approximately 50 to 90% of the total cellulose (Foyle *et al.*, 2004). It is commonly considered as a polymer of glucose because cellobiose consists of two molecules of glucose. The chemical formula of cellulose is $(C_6H_{10}O_5)_n$ and the structure of one chain of the polymer is presented in O.

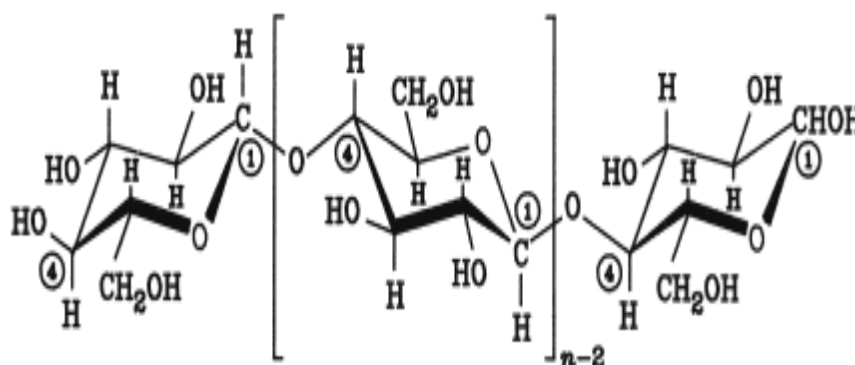


Figure 2.4: Cellulose structure in lignocellulose biomass

2.2.3 Hemicellulose

Hemicellulose is a complex and consists of highly branches polysaccharide. Commonly, the hemicelluloses are located in the secondary cell walls structure together with cellulose and lignin. It is chemically heterogeneous polymer, low degree of polymerization, amorphous and consisting several differences sugars and sugar derivatives. The basic monomeric residues that present are xylose, arabinose, mannose, glucose, galactose, glucuronic acid and methyl ester.

It is mainly composed of xylans that consist of xylose and xylo-ligosaccharides which have highly potential in differences area in chemical food and pharmaceutical industry. Xylan that formed in hemicellulose has a backbone of 1,4- β -linked xylose residues (Figure 2.5). Usually, 75% of the monomers for hemicellulose are pentoses and D-xylose is roughly consist 75% of these sugar. However, hemicellulose has weaker bonding compared to cellulose. It is easily to broken using suitable kinds of pretreatments, such as dilute acid hydrolysis (Saha *et al.*, 2005).

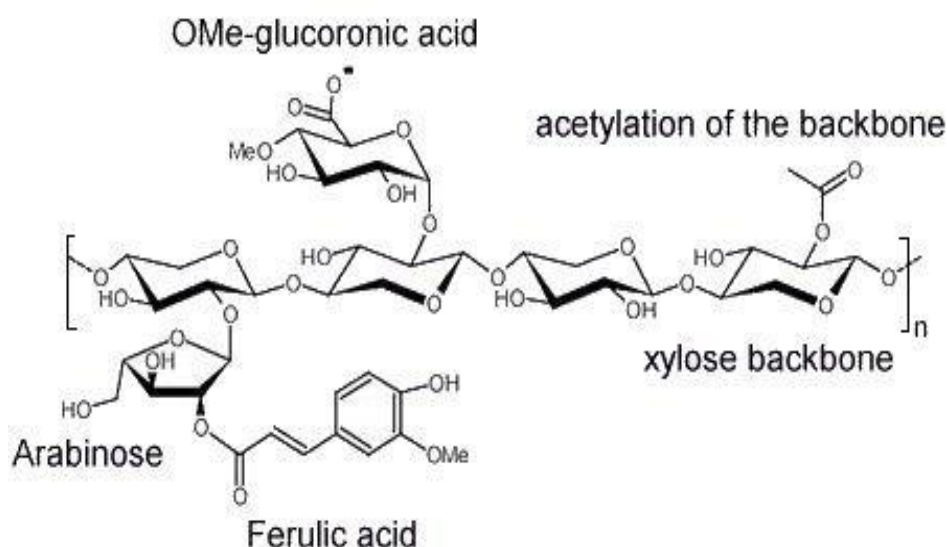


Figure 2.5: Hemicellulose structure in lignocellulose biomass.